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(54) Title: **BLEACHING ACTIVATOR AND PROCESS FOR USING THE ACTIVATOR**

(57) Abstract: The invention relates to a bleaching activator that can be used to improve the opacity of bleached pulps containing lignin, and to a method for using the activator. The activator is mono-, di- or triformic, acetic or propionic ester of glycerol.

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Bleaching activator and process for using the activator

The invention relates to a bleaching activator for improving the opacity of bleached pulps containing lignin, and to a method for using the activator.

- 5 Opacity is used to describe the nontransparent aspect of paper, which, along with brightness, is an important property of pulp in paper manufacture. Almost invariably, however, the opacity of the pulp decreases when the brightness increases. At present, mechanical pulps are more and more often bleached with hydrogen peroxide. Dithionite bleaching is also used either alone or together with peroxide bleaching, whereupon dithionite is either used as refiner bleaching or after-bleaching.
- 10 In the peroxide bleaching of pulp, mechanical pulp in particular, the decrease of opacity is clearly detectable, while the dithionite bleaching does not necessarily decrease the opacity. Generally, the lighter the level of bleaching the pulp, the lower the opacity of the pulp. The appended Fig. 1 that shows a variation in the opacity of
- 15 spruce TMP, when peroxide is used to bleach pulp to various degrees of brightness manifests this. In certain paper grades, opacity is an important property. If we want to advance peroxide bleaching at the expense of dithionite bleaching, it would be important to be able to optimize peroxide bleaching so that the opacity remains as high as possible while the brightness grows.
- 20 Generally, the chemicals used in the peroxide bleaching of mechanical pulps are hydrogen peroxide, lye (alkali), and waterglass. The purpose of the base is to increase the pH to a sufficiently high level, so that the hydrogen peroxide, which works as the actual bleaching agent, is dissociated producing perhydroxyl anions. The purpose of the waterglass is to stabilize the hydrogen peroxide.
- 25 We have observed that peracetic acid treatment, for example, can provide a clearly higher opacity with the same level of brightness than when pulp is bleached with hydrogen peroxide alone.

- Peracetic acid can also be produced in situ, for example, from acetanhydride or TAED (tetra acetyl ethylene diamine) or some other corresponding activator. One
- 30 disadvantage of TAED is its high price and that it is a solid substance. It would be necessary to disperse the TAED in water before use, which makes it difficult to use. Furthermore, TAED contains nitrogen, which might constitute a problem for environmental protection. Acetanhydride is relatively cheap, but it would cause odour nuisance and be an inconvenient substance from the point of view of industrial

safety. In addition, when fed into an alkaline bleaching solution ($\text{NaOH} + \text{H}_2\text{O}_2 + \text{waterglass}$), it would readily cause silicate precipitate and consume the lye.

Paper manufacture aims at ever-higher brightness levels. The brightness of paper can be affected, for example, by treating the paper with coating agents containing, among other things, pigments, binding agents, and plasticizing agents (JP application 284598). However, the use of several coating agents at the final stage of paper manufacture adds to the manufacturing costs.

A technically useful activator should be liquid and stable, and it should preferably have a suitable pH value, so that no silicate precipitate would form in the alkaline peroxide bleaching. Because of environmental matters, a nitrogen-free activator would provide an additional benefit. The additive of the bleaching should also be cost-effective for the paper manufacturers. Consequently, an activator should be found for pulp bleaching, which, to fulfil the conditions mentioned above, is a registered, reasonable, commercial chemical that is easy to get and can be added to the pulp as early as at the peroxide bleaching stage. Furthermore, attention should also be paid to the other effects of the substance, such as applicability in plant conditions.

The purpose of this invention is to find a useful activator that is used in pulp bleaching and that fulfils the conditions mentioned above.

The main features of the invention are disclosed by the appended Claims.

According to the invention, we have surprisingly observed that carboxylic acid esters of glycerol known per se are very suitable to be used as activator agents. From the point of view of industrial hygiene, the carboxylic acid esters of glycerol are almost harmless. Useful carboxylic esters of glycerol include the monoesters, diesters, and triesters of formic acid, acetic acid, and propionic acid in particular. Especially preferable activator agents to be added to the bleaching process comprise acetic esters of glycerol, such as triacetine and diacetine. Even if these esters as such were not water soluble, they dissolve completely in an alkaline peroxide solution, because the acetyl groups split off producing percarboxylic acid in situ. Glycerol and carboxylic acid are the residues of the chemical. By default, bleaching produces glycerol and acetic acid.

Activators according to the invention include mono-, di- and triformic, acetic and propionic esters of glycerol. Mono-, di- and triacetic esters of glycerol are preferable.

As it is assumed that the generation of peracid in peroxide bleaching is the reaction mechanism of the activator, the excess length of the hydrocarbon chain reduces the effect of the activator. When the hydrocarbon chain increases, a smaller amount of peracid is obtained as the amount of material than with shorter hydrocarbon chains.

- 5 A suitable dose of the activator has been found to be 0.2 – 5 kg/ton of pulp. A preferable dosage is 1 – 3 kg/ton of pulp. The bleaching conditions can be normal; in bleaching mechanical pulps, for example, we have used a temperature of 50 - 90°C, a consistency of 5 – 40%, and a retention of 30 – 240 min. Depending on the level of brightness, the dose of peroxide may vary within 5 – 50 kg/ton of pulp. Correspondingly, the doses of lye and waterglass must be adjusted to be suitable for the
10 dose of peroxide. In addition to lye, waterglass, and hydrogen peroxide, the bleaching solution can contain a chelating agent, such as DTPA or some other stabilizers. The activators are suitable to be used for bleaching mechanical pulps in particular, such as ground wood (SGW, PGW), refiner mechanical pulp (TMP) or chemi-
15 mechanical pulps (CTMP). Activators can also be used in the peroxide bleaching of chemical pulps, such as sulphate and sulphite pulp. The sort of wood used for the manufacture of pulp has no significance for the functioning of the invention.

In the following, the invention is described mainly with the aid of examples 1 to 4.

Example 1

- 20 Chemi-mechanical pulp (CTMP) was treated with peroxide in a normal manner. The effect of the bleaching activator is shown in Table 1.

Table 1

CTMP, bleaching solution: 24 kg of NaOH + 20 kg of waterglass + 30 kg/ton of pulp of H₂O₂

- 25 t = 70°C, consistency 30%, 120 min, chelated pulp

| Activator | Dosage, kg/ton of pulp | Brightness, % ISO | Yellowness | Opacity |
|------------|------------------------|-------------------|------------|---------|
| None | - | 78.4 | 17.7 | 64.9 |
| PAA | 2 | 79.1 | 17 | 67.2 |
| Triacetine | 5 | 78 | 17.9 | 67.2 |
| Triacetine | 2 | 78.3 | 17.8 | 68.6 |

The results indicate that by adding the activator to the peroxide bleaching, a distinctly higher opacity with the same brightness level is achieved than by using peroxide bleaching alone. The results also show that the activators had hardly any effect on the ISO brightness.

5 Example 2

Refiner mechanical pulp of spruce with a brightness of 60.4% ISO, opacity of 86.5, containing 100 ppm of Mn and 18 ppm of Fe, was brought to peroxide bleaching. The results are in Table 2.

Table 2 TMP (spruce), bleaching stages

- 10 Chelating treatment: Consistency 10%, pH 5.5, 45 min, $t = 55^{\circ}\text{C}$, 2 kg/ton of pulp of DTPA

Consistency to 15%

Peroxide bleaching: 120 min, $t = 70^{\circ}\text{C}$, consistency 15%, 22 kg of H_2O_2 , 22 kg of NaOH, 17.6 kg/ton of pulp of waterglass

| Activator | Dosage kg/ton of pulp | H_2O_2 residue, kg/ton of pulp | Brightness, % ISO | Opacity, % |
|------------|-----------------------|--|-------------------|------------|
| None | 0 | 9.2 | 74.5 | 81.4 |
| Triacetine | 1 | 9.4 | 75.8 | 83.1 |
| Triacetine | 2 | 9.9 | 75.5 | 82.8 |

15

The results show that the activator in peroxide bleaching obviously had a positive effect on the opacity of the pulp, when compared with peroxide bleaching without the added activator.

Example 3

- 20 Pressure groundwood pulp was treated with peroxide in a normal manner. The effect of the bleaching activator is shown in Table 3.

Table 3 PGW (pressure groundwood pulp)

Peroxide bleaching: Consistency 28%, 12 0min, $t = 70^{\circ}\text{C}$, 25 kg of H_2O_2 , 18.8 kg of waterglass, 25 kg of NaOH, chelated at the plant

| Triacetine, kg/ton of pulp | Brightness, % ISO | Opacity, % | Light scatter- ing | Light absorp- tion |
|-------------------------------|----------------------|------------|-----------------------|-----------------------|
| 0 | 77.5 | 86.4 | 67.4 | 0.37 |
| 1 | 77.8 | 87.8 | 70.3 | 0.36 |
| 2 | 77.8 | 88.6 | 73.9 | 0.37 |

5 Example 4

Mechanical pulp was treated with peroxide in a normal manner. The effect of the bleaching activator in peroxide bleaching is shown in Table 4.

Table 4

10 TMP, bleaching solution: 20 kg of NaOH + 18.8 kg of waterglass + 20 kg of H_2O_2 , 2 kg of DTPA

$t = 70^{\circ}\text{C}$, consistency 28%, 120 min, plant-chelated pulp

| Activator | Dosage, kg/ton of pulp | Brightness, % ISO | Opacity |
|---------------|------------------------|-------------------|---------|
| None | - | 77.8 | 79.4 |
| Triacetine | 1 | 77.9 | 81.3 |
| 15 Triacetine | 2 | 77.8 | 81.1 |
| Triacetine | 5 | 77.5 | 81.7 |
| Diacetine | 2 | 77.8 | 81.5 |

20 The results show that the activator had a distinct effect on the opacity with the same level of brightness as peroxide bleaching alone. We can also observe that the activators have no effect on the ISO brightness.

Claims

1. A bleaching activator for use in paper pulp containing lignin, characterized in that the activator is a mono-, di- or triformic, acetic or propionic ester of glycerol.
2. A bleaching activator according to Claim 1, characterized in that the activator
5 is glycerol triacetate.
3. A bleaching activator according to Claim 1, characterized in that the activator is glycerol diacetate.
4. A bleaching activator according to Claim 1, characterized in that the activator is used in peroxide bleaching of chemical or mechanical pulp.
- 10 5. A bleaching activator according to Claim 1, characterized in that the amount of the activator used is 0.2 – 5kg/ton of pulp.
6. A bleaching activator according to Claim 1, characterized in that the amount of the activator used is 1 – 3kg/ton of pulp.
7. A bleaching activator according to Claim 1, characterized in that the bleach-
15 ing solution containing the activator also contains chelating agents, stabilizers, lye, and waterglass.
8. A bleaching activator according to Claim 1, characterized in that the activator is used together with peracetic acid.
9. The use of mono-, di- or triformic, acetic or propionic ester of glycerol as a
20 bleaching activator in paper pulp containing lignin.
10. A bleaching method for pulp containing lignin, characterized in that an activator according to Claim 1 is added to a bleaching solution containing hydrogen peroxide to improve the opacity.

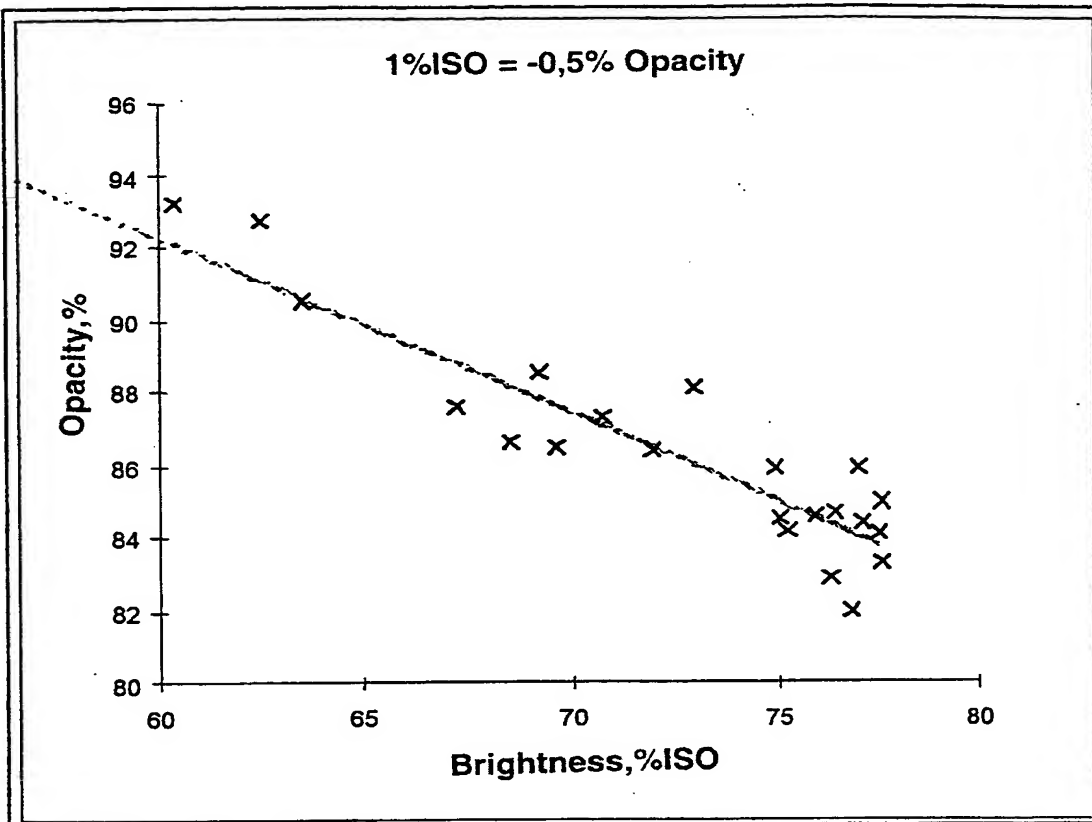


Fig. 1

INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 00/00534

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: D21C 9/16

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: D21C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|-----------|---|-----------------------|
| X | EP 0481792 A1 (UNILEVER NV), 22 April 1992 (22.04.92), page 3, line 47 - line 49, claims 1,5 -- | 1-9 |
| A | WO 9521290 A1 (WARWICK INTERNATIONAL GROUP LIMITED), 10 August 1995 (10.08.95), claim 1 -- | 1-10 |
| A | WO 9418299 A1 (WARWICK INTERNATIONAL GROUP LIMITED), 18 August 1994 (18.08.94) -- | 1-10 |

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

* Special categories of cited documents

"A" document defining the general state of the art which is not considered to be of particular relevance

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"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

26 October 2000

Date of mailing of the international search report

01-11-2000

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 00/00534

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/FI 00/00534

| Patent document cited in search report | | | Publication date | Patent family member(s) | Publication date |
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PCT REQUEST

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| 0 0-1 | For receiving Office use only International Application No. | PCT/FI 0 0 / 0 0 5 3 4 |
| 0-2 | International Filing Date | 1 4 JUN 2000 (1 4 -06- 2000) |
| 0-3 | Name of receiving Office and "PCT International Application" | The Finnish Patent Office PCT International Application |
| 0-4 0-4-1 | Form - PCT/RO/101 PCT Request Prepared using | PCT-EASY Version 2.90 (updated 10.05.2000) |
| 0-5 | Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty | |
| 0-6 | Receiving Office (specified by the applicant) | National Board of Patents and Registration (Finland) (RO/FI) |
| 0-7 | Applicant's or agent's file reference | 50136 |
| I | Title of invention | BLEACHING ACTIVATOR AND PROCESS FOR USING THE ACTIVATOR |
| II II-1 II-2 II-4 II-5 | Applicant This person is: Applicant for Name Address: | applicant only all designated States except US KEMIRA CHEMICALS OY P.O. Box 330 FIN-00101 Helsinki Finland |
| II-6 | State of nationality | FI |
| II-7 | State of residence | FI |
| III-1 III-1-1 III-1-2 III-1-4 III-1-5 | Applicant and/or inventor This person is: Applicant for Name (LAST, First) Address: | applicant and inventor US only JÄKÄRÄ, Jukka Koulukatu 32 A 10 FIN-65100 Vaasa Finland |
| III-1-6 | State of nationality | FI |
| III-1-7 | State of residence | FI |


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| | | |
|---------|--|---|
| III-2 | Applicant and/or inventor | |
| III-2-1 | This person is: | applicant and inventor |
| III-2-2 | Applicant for | US only |
| III-2-4 | Name (LAST, First) | PARÉN, Aarto |
| III-2-5 | Address: | Kapteeninkatu 36 B 20 FIN-65200 Vaasa Finland |
| III-2-6 | State of nationality | FI |
| III-2-7 | State of residence | FI |
| IV-1 | Agent or common representative; or address for correspondence | |
| | The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: | agent |
| IV-1-1 | Name | BERGGREN OY AB |
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| IV-1-3 | Telephone No. | +358-9-693701 |
| IV-1-4 | Facsimile No. | +358-9-6933944 |
| IV-1-5 | e-mail | email.box@berggren.fi |
| V | Designation of States | |
| V-1 | Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) | AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT EP: AT BE CH&LI CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE and any other State which is a Contracting State of the European Patent Convention and of the PCT OA: BF BJ CF CG CI CM GA GN GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT |
| V-2 | National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) | AE AG AL AM AT AU AZ BA BB BG BR BY CA CH&LI CN CR CU CZ DE DK DM DZ EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US UZ VN YU ZA ZW |

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|---------|---|--|
| V-5 | Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. | |
| V-6 | Exclusion(s) from precautionary designations | NONE |
| VI-1 | Priority claim of earlier national application | |
| VI-1-1 | Filing date | 15 June 1999 (15.06.1999) |
| VI-1-2 | Number | 991365 |
| VI-1-3 | Country | FI |
| VI-2 | Priority document request The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) identified above as item(s): | VI-1 |
| VII-1 | International Searching Authority Chosen | Swedish Patent Office (ISA/SE) |
| VIII | Check list | number of sheets electronic file(s) attached |
| VIII-1 | Request | 4 - |
| VIII-2 | Description | 5 - |
| VIII-3 | Claims | 1 - |
| VIII-4 | Abstract | 1 50136.txt |
| VIII-5 | Drawings | 1 - |
| VIII-7 | TOTAL | 12 |
| | Accompanying items | paper document(s) attached electronic file(s) attached |
| VIII-8 | Fee calculation sheet | ✓ - |
| VIII-10 | Copy of general power of attorney | ✓ - |
| VIII-16 | PCT-EASY diskette | - diskette |
| VIII-18 | Figure of the drawings which should accompany the abstract | |
| VIII-19 | Language of filing of the international application | Finnish |
| IX-1 | Signature of applicant or agent |  |
| IX-1-1 | Name | BERGGREN OY AB |
| IX-1-2 | Name of signatory | Berndt Traskman |
| IX-1-3 | Capacity | Patent Attorney |

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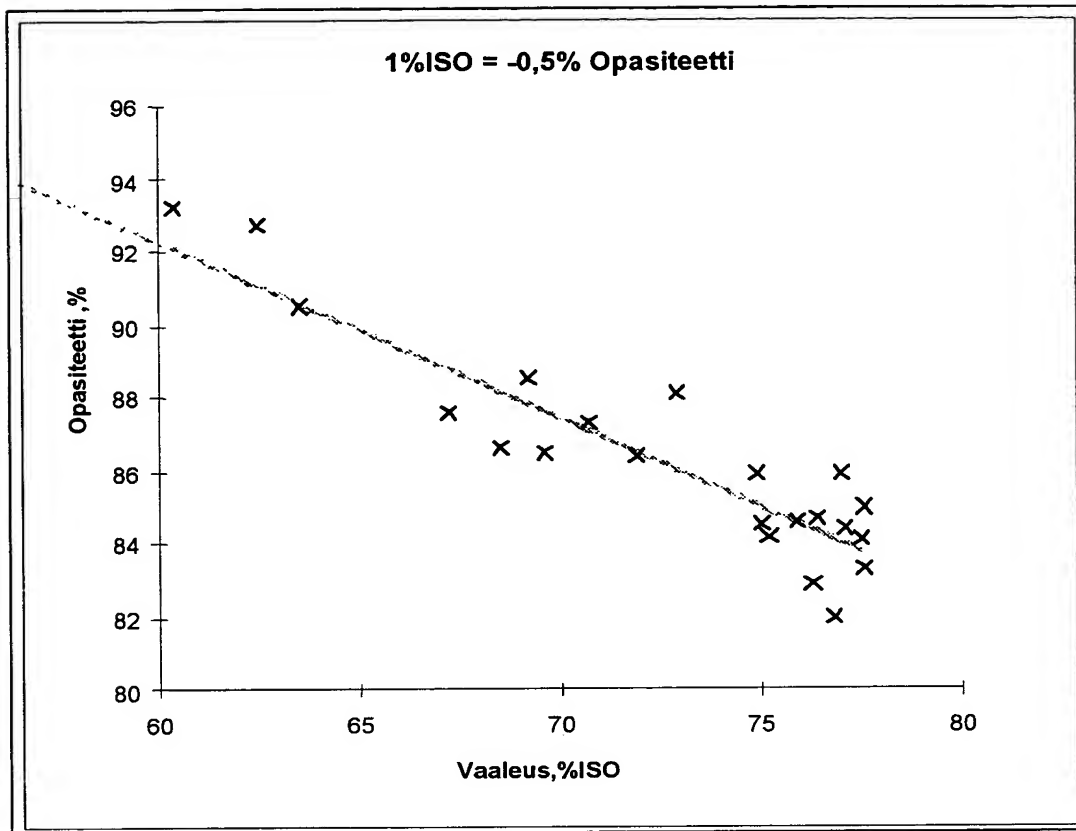
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|------|---|-------------|--------------|
| 10-1 | Date of actual receipt of the purported international application | 14 JUN 2000 | (14-06-2000) |
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| | | |
|--------|---|--------|
| 10-2 | Drawings: | |
| 10-2-1 | Received | |
| 10-2-2 | Not received | |
| 10-3 | Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application | |
| 10-4 | Date of timely receipt of the required corrections under PCT Article 11(2) | |
| 10-5 | International Searching Authority | ISA/SE |
| 10-6 | Transmittal of search copy delayed until search fee is paid | |

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|------|--|--------------|-----------------|
| 11-1 | Date of receipt of the record copy by the International Bureau | 03 JULY 2000 | (0 3. 07. 00) |
|------|--|--------------|-----------------|



Kuva 1

Valkaisuaktivaattori ja menetelmä aktivaattorin käyttämiseksi

Keksintö koskee valkaisuaktivaattoria, jolla voidaan parantaa valkaistujen, ligniiniä sisältävien massojen opasiteettia, ja menetelmää aktivaattorin käyttämiseksi.

- 5 Paperin läpikuultamattomuutta kuvaa opasiteetti, joka on vaaleuden ohella tärkeä massan ominaisuus paperin valmistuksessa. Lähes poikkeuksetta massan opasiteetti kuitenkin laskee vaaleuden noustessa. Nykyisin mekaanisia massoja valkaistaan yhä useammin vetyperoksidilla. Myös ditioniittivalkaisua käytetään, joko yhdessä tai yksin peroksidivalkaisun kanssa, jolloin ditioniittia käytetään joko jauhinvalkaisuna tai jälkivalkaisuna. Massojen, etenkin mekaanisten massojen peroksidivalkaisussa opasiteetin lasku havaitaan selvästi, kun taas ditioniittivalkaisu ei välttämättä alenna opasiteettia. Yleensä massan opasiteetti laskee sitä enemmän, mitä vaaleammaksi massa valkaistaan. Tämä ilmenee oheisesta kuvasta 1, joka esittää kuusi-TMp:n opasiteetin muutosta valkaistaessa massaa peroksidilla eri vaaleustasoille. Tietyissä paperilaaduissa opasiteetti on tärkeä ominaisuus. Kun halutaan edistää peroksidivalkaisua ditioniittivalkaisun kustannuksella, olisi tärkeää pystyä optimoimaan peroksidivalkaisu niin, että opasiteetti säilyisi mahdollisimman korkeana vaaleuden kas-
- 10
15

- 20 Mekaanisten massojen peroksidivalkaisussa käytetyt kemikaalit ovat yleisimmin vetyperoksidi, lipeä (alkali) ja vesilasi. Emäksen tarkoituksena on nostaa pH riittävän korkealle, jotta varsinaisena valkaisevana aineena toimiva vetyperoksidi dissoioituu tuottaen perhydroksyylianioneja. Vesilasin tarkoituksena on stabiloida vetyperoksidia.

- 25 On havaittu, että esimerkiksi peretikkahappokäsittelyllä voidaan saada aikaan selvästi korkeampi opasiteetti samalla vaaleustasolla kuin pelkällä vetyperoksidilla valkaistulla massalla.

- 30 Peretikkahappoa voidaan muodostaa myös in situ esimerkiksi asetanhydridistä tai TAED:sta (tetra-asetyyli-etyleen-diämiini) tai jostakin muusta vastaavasta aktivaattorista. TAED:n haittana on sen korkea hinta ja se, että se on kiinteä aine. Käyttöä varten TAED jouduttaisiin dispergoimaan veteen ennen käyttöä, mikä tekee sen käytön hankalaksi. Lisäksi TAED sisältää typpeä, mikä voi tulla ongelmaksi ympäristön suojelun kannalta. Asetanhydridi olisi suhteellisen halpaa, mutta se aiheuttaisi hajuhaittoja ja olisi työsuojelun kannalta hankala aine. Lisäksi alkaliseen valkaisu-

liuokseen ($\text{NaOH} + \text{H}_2\text{O}_2 + \text{vesilasi}$) syötettynä se aiheuttaisi helposti silikaattisaostumia ja kuluttaisi lipeää.

5 Paperinvalmistuksessa tähdätään yhä korkeampiin vaaleuksiin. Paperin vaaleuteen voidaan vaikuttaa esimerkiksi käsittelemällä paperia päällystysaineilla, jotka sisältävät mm. pigmenttejä, sideaineita ja pehmitinaineita (JP-hakemus 284598). Kuitenkin useiden päällystysaineiden käyttö paperinvalmistuksen loppuvaiheessa lisää edelleen valmistuskustannuksia.

10 Teknisesti käyttökelpoisen aktivaattorin tulisi olla nestemäinen, pysyvä ja mieluiten pH-arvoltaan sopiva, jottei alkalisessa peroksidivalkaisussa syntyisi silikaattisaostumia. Lisäetuna olisi typetön aktivaattori ympäristönsuojelullisista syistä. Valkaisun apuaineen tulee myös olla edullista paperinvalmistajille. Massojen valkaisuun on siis löydettävä aktivaattori, joka edellä mainittujen ehtojen täyttämiseksi on rekisteröity ja helposti saatavissa oleva kohtuuhintainen kaupallinen kemikaali ja joka voidaan lisätä massaan jo peroksidivalkaisuvaiheessa. Lisäksi on myös kiinnitettävä
15 huomiota aineen muihin vaikutuksiin, kuten käyttökelpoisuuteen tehdasoloissa.

Tämän keksinnön tarkoituksena on löytää käyttökelpoinen massan valkaisussa käytettävä aktivaattori, joka täyttää edellä mainitut vaatimukset.

Keksinnön pääasialliset tunnusmerkit ilmenevät oheisista patenttivaatimuksista.

20 Keksinnön mukaisesti on yllättäen havaittu, että sinänsä tunnetut glyserolin karboksyylihappoesterit ovat sangen sopivia aktivaattoriaineiksi. Glyserolin karboksyylihappoesterit ovat työhygienian kannalta lähes haitattomia. Glyserolin karboksyyliesteriestä käyttökelpoisia ovat erityisesti muurahais-, etikka- ja propionihapon mono-, di- ja triesterit. Erityisen edullisia lisättäviä aktivaattoriaineita valkaisuprosessiin ovat glyserolin etikkahappoesterit, mm. triasetiini ja diasetiini. Vaikka nämä esterit
25 eivät jo sellaisenaan olisi vesiliukoisia, liukenevat ne täysin alkaliseen peroksidiliuokseen, koska asetyyli-ryhmät lohkeavat irti tuottaen perkarboksyylihappoa in situ. Jäännöksenä kemikaalista on glyseroli ja karboksyylihappo. Valkaisussa syntyy luonnostaankin glyserolia ja etikkahappoa.

30 Keksinnön mukaisia aktivaattoreita ovat glyserolin muurahais-, etikka- ja propionihappojen mono-, di- ja triesterit. Edullisia ovat glyserolin etikkahapon mono-, di- ja triesterit.

Hiilivetyketjun liiallinen pituus vähentää aktivaattorin tehoa, sillä aktivaattorin reaktiomekanismiksi on oletettu perhapon syntyminen peroksidivalkaisussa. Hiilivety-

ketjun kasvaessa saadaan ainemääränä vähäisempi määrä perhappoa kuin lyhyemmillä hiilivetyketjuilla.

- Sopivaksi aktivaattoriannokseksi on havaittu 0,2-5 kg/massatonni. Edullinen annostus on 1-3 kg/massatonni. Valkaisuolosuhteet voivat olla normaalit, mm. mekaanisten massojen valkaisuissa on käytetty lämpötilaa 50-90 °C, sakeutta 5-40 % ja viipymää 30-240 minuuttia. Peroksidiannos voi vaihdella vaaleustavoitteesta riippuen 5-50 kg/massatonni. Vastaavasti lipeä- ja vesilasiannokset on sovitettava peroksidiannoon sopiviksi. Valkaisuliouksessa voi lipeän, vesilasin ja vetyperoksidin lisäksi olla kelatointiaineita, kuten DTPA:ta tai joitakin muita stabilaattoreita. Aktivaattorit soveltuvat käytettäväksi erityisesti mekaanisten massojen valkaisuun, kuten hiokkeen (SGW, PGW), hierteen (TMP) tai kemimekaanisten massojen (CTMP) valkaisuun. Aktivaattoreita voidaan myös käyttää kemiallisten massojen, kuten sulfaatti- ja sulfiittisellun peroksidivalkaisussa. Massan valmistukseen käytetyllä puulajilla ei ole merkitystä keksinnön toimivuuden kannalta.
- Seuraavassa keksintöä selostetaan lähinnä esimerkkien 1-4 avulla.

Esimerkki 1

Kemimekaanista-massaa (CTMP) käsiteltiin normaaliin tapaan peroksidilla. Valkaisuaktivaattorin vaikutus on kuvattu taulukossa 1.

Taulukko 1

- CTMP, valkaisuliuos: NaOH 24 kg + vesilasi 20 kg + H₂O₂ 30 kg/tm
t = 70 °C, sakeus 30 %, 120 min, kelatoitu massa

| Aktivaattori | Annos, kg/tm | Vaaleus, % ISO | Keltaisuus | Opasiteetti |
|--------------|-----------------|-------------------|------------|-------------|
| Ei | - | 78,4 | 17,7 | 64,9 |
| PAA | 2 | 79,1 | 17 | 67,2 |
| Triasetiini | 5 | 78 | 17,9 | 67,2 |
| Triasetiini | 2 | 78,3 | 17,8 | 68,6 |

- Tuloksista havaittiin, että aktivaattorilisäyksellä peroksidivalkaisuun saadaan aikaan selvästi korkeampi opasiteetti samalla vaaleustasolla kuin pelkällä peroksidivalkaisulla. Tuloksista voidaan myös havaita, etteivät aktivaattorit juurikaan vaikuttaneet ISO-vaaleuteen.

Esimerkki 2

Kuusihierrmassaa, jonka vaaleus oli 60,4 % ISO, opasiteetti 86,5 ja joka sisälsi Mn 100 ppm ja Fe 18 ppm, saatettiin peroksidivalkaisuun. Tulokset ovat taulukossa 2.

Taulukko 2 TMP (kuusi) valkaisuvaiheet

Kelatointikäsitely: Sakeus 10 %, pH 5,5, 45 min, t = 55 °C, DTPA 2 kg/tm

Sakeutus 15 %:iin

Peroksidivalkaisu: 120 min, t = 70 °C, sakeus 15 %, H₂O₂ 22 kg, NaOH 22 kg, vesilasi 17,6 kg/tm

| Aktivaattori | Annos kg/tm | Jäännös- H ₂ O ₂ , kg/tm | Vaaleus, % ISO | Opasiteetti, % |
|--------------|----------------|---|-------------------|-------------------|
| Ei | 0 | 9,2 | 74,5 | 81,4 |
| Triasetiini | 1 | 9,4 | 75,8 | 83,1 |
| Triasetiini | 2 | 9,9 | 75,5 | 82,8 |

- 5 Tuloksista havaittiin aktivaattorilla olevan peroksidivalkaisussa selvä positiivinen vaikutus massan opasiteettiin, kun vertailuna oli peroksidivalkaisu ilman aktivaattorilisäystä.

Esimerkki 3

Painehiokemassaa käsiteltiin normaaliin tapaan peroksidilla. Valkaisuaktivaattorin vaikutus on kuvattu taulukossa 3.

Taulukko 3 PGW (painehioke)

Peroksivalkaisu: sakeus 28 %, 120 min, t = 70 °C, H₂O₂ 25 kg, vesilasi 18,8 kg, NaOH 25 kg, tehdaskelatoitu

| Triasetiini, kg/tm | Vaaleus, % ISO | Opasiteetti, % | Valonsironta | Valonabsorptio |
|-----------------------|-------------------|----------------|--------------|----------------|
| 0 | 77,5 | 86,4 | 67,4 | 0,37 |
| 1 | 77,8 | 87,8 | 70,3 | 0,36 |
| 2 | 77,8 | 88,6 | 73,9 | 0,37 |

Esimerkki 4

Mekaanista massaa käsiteltiin normaaliin tapaan peroksidilla. Valkaisuaktivaattorin vaikutus peroksidivalkaisussa on kuvattu taulukossa 4.

Taulukko 4 TMP, valkaisuliuos: NaOH 20 kg + vesilasi 18,8 kg + H₂O₂
20 kg, DTPA 2 kg

t = 70 °C, sakeus 28 %, 120 min, tehdaskelatoitu massa

| Aktivaattori | Annos, Vaaleus,% | | Opasiteetti |
|--------------|------------------|------|-------------|
| | kg/tm | ISO | |
| Ei | - | 77,8 | 79,4 |
| Triasetiini | 1 | 77,9 | 81,3 |
| Triasetiini | 2 | 77,8 | 81,1 |
| Triasetiini | 5 | 77,5 | 81,7 |
| Diasetiini | 2 | 77,8 | 81,5 |

- 5 Tuloksista havaittiin selvä aktivaattorin vaikutus opasiteettiin samalla vaaleustasolla kuin pelkällä peroksidivalkaisulla. Samoin voidaan havaita, että aktivaattorit eivät vaikuta ISO-vaaleuteen.

Patenttivaatimukset

1. Ligniiniä sisältävässä paperimassassa käytettävä valkaisuaktivaattori, tunnettu siitä, että aktivaattori on glyserolin muurahais-, etikka- tai propionihapon mono-, di- tai triesteri.
- 5 2. Patenttivaatimuksen 1 mukainen valkaisuaktivaattori, tunnettu siitä, että aktivaattori on glyserolitriasetaatti.
3. Patenttivaatimuksen 1 mukainen valkaisuaktivaattori, tunnettu siitä, että aktivaattori on glyserolidiasetaatti.
- 10 4. Patenttivaatimuksen 1 mukainen valkaisuaktivaattori, tunnettu siitä, että aktivaattoria käytetään kemiallisen tai mekaanisen massan peroksidivalkaisussa.
5. Patenttivaatimuksen 1 mukainen valkaisuaktivaattori, tunnettu siitä, että aktivaattoria käytetään 0,2-5 kg/massatonni.
6. Patenttivaatimuksen 1 mukainen valkaisuaktivaattori, tunnettu siitä, että aktivaattoria käytetään 1-3 kg/massatonni.
- 15 7. Patenttivaatimuksen 1 mukainen valkaisuaktivaattori, tunnettu siitä, että aktivaattoria sisältävä valkaisuliuos lisäksi sisältää kelatointiaineita, stabilaattoreita, lipeää ja vesilasia.
8. Patenttivaatimuksen 1 mukainen valkaisuaktivaattori, tunnettu siitä, että aktivaattoria käytetään yhdessä peretikkahapon kanssa.
- 20 9. Glyserolin muurahais-, etikka- tai propionihapon mono-, di- tai triesterin käyttö valkaisuaktivaattorina ligniiniä sisältävässä paperimassassa.
10. Ligniiniä sisältävän massan valkaisumenetelmä, tunnettu siitä, että vetyperoksidia sisältävään valkaisuliuokseen lisätään opasiteetin parantamiseksi vaatimuksen 1 mukaista aktivaattoria.

(57) Tiivistelmä

Keksintö koskee valkaisuaktivaattoria, jolla voidaan parantaa valkaistujen ligniiniä sisältävien massojen opasiteettia, ja menetelmää sen käyttämiseksi. Aktivaattori on glyserolin muurahais-, etikka- tai propionihapon mono-, di- tai triesteri.

PATENT COOPERATION TREATY

PCT

NOTICE INFORMING THE APPLICANT OF THE
COMMUNICATION OF THE INTERNATIONAL
APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

From the INTERNATIONAL BUREAU

To:

BERGGREN OY AB
P.O. Box 16
FIN-00101 Helsinki
FINLANDE*Berggren Oy Ab*

02-01-2001

JS/BL

| | | | |
|---|---|---|--|
| Date of mailing (day/month/year) 21 December 2000 (21.12.00) | | IMPORTANT NOTICE | |
| Applicant's or agent's file reference 50136 | | | |
| International application No. PCT/FI00/00534 | International filing date (day/month/year) 14 June 2000 (14.06.00) | Priority date (day/month/year) 15 June 1999 (15.06.99) | |
| Applicant KEMIRA CHEMICALS OY et al | | | |

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:

AG,AU,DZ,KP,KR,MZ,US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

AE,AL,AM,AP,AT,AZ,BA,BB,BG,BR,BY,CA,CH,CN,CR,CU,CZ,DE,DK,DM,EA,EE,EP,ES,FI,GB,GD,
GE,GH,GM,HR,HU,ID,IL,IN,IS,JP,KE,KG,KZ,LC,LK,LR,LS,LT,LU,LV,MA,MD,MG,MK,MN,MW,MX,
NO,NZ,OA,PL,PT,RO,RU,SD,SE,SG,SI,SK,SL,TJ,TM,TR,TT,TZ,UA,UG,UZ,VN,YU,ZA,ZW
The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on 21 December 2000 (21.12.00) under No. WO 00/77297

REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a demand for international preliminary examination must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the national phase, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

| | |
|--|---|
| The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No. (41-22) 740.14.35 | Authorized officer J. Zahra Telephone No. (41-22) 338.83.38 |
|--|---|

Continuation of Form PCT/IB/308

**NOTICE INFORMING THE APPLICANT OF THE COMMUNICATION OF
THE INTERNATIONAL APPLICATION TO THE DESIGNATED OFFICES**

| | |
|---|--|
| Date of mailing (day/month/year) 21 December 2000 (21.12.00) | IMPORTANT NOTICE |
| Applicant's or agent's file reference 50136 | International application No. PCT/FI00/00534 |
| <p>The applicant is hereby notified that, at the time of establishment of this Notice, the time limit under Rule 46.1 for making amendments under Article 19 has not yet expired and the International Bureau had received neither such amendments nor a declaration that the applicant does not wish to make amendments.</p> | |

PARENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION CONCERNING
SUBMISSION OR TRANSMITTAL
OF PRIORITY DOCUMENT

(PCT Administrative Instructions, Section 411)

To:

BERGGREN OY AB
P.O. Box 16
FIN-00101 Helsinki
FINLANDE*Berggren Oy Ab*

18-10-2000

DS/RL

| | |
|--|---|
| Date of mailing (day/month/year) 09 October 2000 (09.10.00) | IMPORTANT NOTIFICATION |
| Applicant's or agent's file reference 50136 | |
| International application No. PCT/FI00/00534 | International filing date (day/month/year) 14 June 2000 (14.06.00) |
| International publication date (day/month/year) Not yet published | Priority date (day/month/year) 15 June 1999 (15.06.99) |
| Applicant KEMIRA CHEMICALS OY et al | |

- The applicant is hereby notified of the date of receipt (except where the letters "NR" appear in the right-hand column) by the International Bureau of the priority document(s) relating to the earlier application(s) indicated below. Unless otherwise indicated by an asterisk appearing next to a date of receipt, or by the letters "NR", in the right-hand column, the priority document concerned was submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b).
- This updates and replaces any previously issued notification concerning submission or transmittal of priority documents.
- An asterisk(*) appearing next to a date of receipt, in the right-hand column, denotes a priority document submitted or transmitted to the International Bureau but not in compliance with Rule 17.1(a) or (b). In such a case, **the attention of the applicant is directed to Rule 17.1(c)** which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.
- The letters "NR" appearing in the right-hand column denote a priority document which was not received by the International Bureau or which the applicant did not request the receiving Office to prepare and transmit to the International Bureau, as provided by Rule 17.1(a) or (b), respectively. In such a case, **the attention of the applicant is directed to Rule 17.1(c)** which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.

| <u>Priority date</u> | <u>Priority application No.</u> | <u>Country or regional Office or PCT receiving Office</u> | <u>Date of receipt of priority document</u> |
|-------------------------|---------------------------------|---|---|
| 15 June 1999 (15.06.99) | 991365 | FI | 28 Augu 2000 (28.08.00) |

| | |
|--|---|
| The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No. (41-22) 740.14.35 | Authorized officer Somsak Thiphrakesone Telephone No. (41-22) 338.83.38 |
|--|---|

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Rule 71.1)

| | |
|--|--|
| <p>To:</p> <p>BERGGREN OY P.O.Box 16 00101 Helsinki FINLANDE</p> | <p><i>Berggren Oy Ab</i></p> <p>17 -09- 2001</p> <p><i>JS/BLK</i></p> |
|--|--|

| | |
|-------------------------------------|------------|
| Date of mailing (day/month/year) | 11.09.2001 |
|-------------------------------------|------------|

| | |
|---|-------------------------------|
| Applicant's or agent's file reference 50136/JS | IMPORTANT NOTIFICATION |
|---|-------------------------------|

| | | |
|---|--|--|
| International application No. PCT/FI00/00534 | International filing date (day/month/year) 14/06/2000 | Priority date (day/month/year) 15/06/1999 |
|---|--|--|

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|--|
| Applicant KEMIRA CHEMICALS OY et al |
|--|

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/

 European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer

Lázaro Ortiz, A

Tel. +49 89 2399-8011



PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

| | | |
|--|---|--|
| Applicant's or agent's file reference 50136/JS | FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416) | |
| International application No. PCT/FI00/00534 | International filing date (day/month/year) 14/06/2000 | Priority date (day/month/year) 15/06/1999 |
| International Patent Classification (IPC) or national classification and IPC D21C9/16 | | |
| Applicant KEMIRA CHEMICALS OY et al | | |


1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 5 sheets, including this cover sheet.

☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

 These annexes consist of a total of 1 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☒ Certain defects in the international application
- VIII ☒ Certain observations on the international application

| | |
|---|---|
| Date of submission of the demand 14/12/2000 | Date of completion of this report 11.09.2001 |
| Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 | Authorized officer Nestby, K Telephone No. +49 89 2399 8625 |



INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/FI00/00534

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, pages:

1-5 as originally filed

Claims, No.:

1-10 as received on 23/05/2001 with letter of 23/05/2001

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/FI00/00534

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | | | |
|-------------------------------|------|--------|------|
| Novelty (N) | Yes: | Claims | 1-10 |
| | No: | Claims | |
| Inventive step (IS) | Yes: | Claims | 1-10 |
| | No: | Claims | |
| Industrial applicability (IA) | Yes: | Claims | 1-10 |
| | No: | Claims | |

2. Citations and explanations see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. It is known in the art, e. g. from WO-A-94/18299 and WO-A-95/21290 (hereafter referred to as document D2, respectively D3) to bleach cellulose pulp with compositions comprising peroxide as well as bleaching activators. The particular activators of the present invention, however, are not mentioned.

The subject-matter of all claims 1 to 10 is therefore novel (Article 33(2) PCT).

2. Although the use of glycerol triacetate as bleaching activator is described in EP-A-0 481 792 (D1), there is no incentive for the skilled person to use this particular activator in a bleaching composition as disclosed in D1 and/or D2, for the following reasons:

The specific preferred activator used in D3 (closest prior art) is tetraacetylene diamine (TAED). The drawbacks of TAED are discussed in the description of the present application on page 1, line 28 to page 2, line 2.

Said drawbacks are solved by the present invention; besides, a cellulose pulp treated with said composition becomes improved opacity. There is no hint whatsoever in D1 that a detergent composition in tablet form (disclosure of D3) could possibly solve said problems and at the same time bring about an improvement of cellulose pulp opacity properties.

The solution to this problem proposed in claims 1, 6, 7 of the present application is hence considered as involving an inventive step (Article 33(3) PCT). The subject matter of dependent claims 2 to 5, 8 to 10 represents preferred embodiments of the novel and inventive subject matter of claims 1, 7.

Re item VII

Certain defects in the international application

3. Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1, D2, D3 is not mentioned in the description, nor

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/FI00/00534

are these documents identified therein.

4. The description is not in conformity with the amended claims as required by Rule 5.1(a)(iii) PCT.

Re Item VIII

Certain observations on the international application

5. Although claims 1, 7 are drafted in the two-part form the feature "bleaching solution comprises a bleaching activator" is incorrectly placed in the characterising portion, as it is disclosed in documents D1, D2 in combination with the features placed in the preamble (Rule 6.3(b) PCT).

Claims

1. A peroxide bleaching solution for bleaching paper pulp containing lignin, characterized in that said bleaching solution comprises a bleaching activator comprising a mono-, di- or triformic, acetic or propionic ester of glycerol.
2. A peroxide bleaching solution according to Claim 1, wherein the activator is glycerol triacetate.
3. A peroxide bleaching solution according to Claim 1, wherein the activator is glycerol diacetate.
4. A peroxide bleaching solution according to Claim 1, wherein the bleaching solution additionally comprises a chelating agent, a stabilizer, lye, and waterglass.
5. A peroxide bleaching solution according to Claim 1, wherein the bleaching solution comprises peracetic acid.
6. Use of mono-, di- or triformic, acetic or propionic ester of glycerol as a bleaching activator in peroxide bleaching of paper pulp containing lignin to improve the opacity.
7. A method for bleaching pulp containing lignin with a bleaching solution containing hydrogen peroxide, characterized in that a bleaching activator comprising a mono-, di- or triformic, acetic or propionic ester of glycerol is added to the bleaching to improve the opacity.
8. A method according to claim 7, wherein the pulp to be bleached comprises a mechanical pulp.
9. A method according to Claim 7, wherein the activator is added in an amount of 0.2 - 5 kg/ton of pulp.
10. A method according to Claim 7, wherein the activator is added in an amount of 1 - 3 kg/ton of pulp.

PATENT COOPERATION TREATY

From the:
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

BERGGREN OY
P.O.Box 16
00101 Helsinki
FINLANDE

Berggren Oy Ab
28-03-2001

JS/BK

PCT

WRITTEN OPINION

(PCT. Rule 66)

| | | |
|--|---|---|
| Applicant's or agent's file reference 50136/JS | | Date of mailing (day/month/year) 26.03.2001 |
| International application No. PCT/FI00/00534 | | REPLY DUE within 2 month(s) from the above date of mailing. <i>26.5</i> |
| International filing date (day/month/year) 14/06/2000 | Priority date (day/month/year) 15/06/1999 | |
| International Patent Classification (IPC) or both national classification and IPC D21C9/16 | | |
| Applicant KEMIRA CHEMICALS OY et al | | |

1. This written opinion is the **first** drawn up by this International Preliminary Examining Authority.
2. This opinion contains indications relating to the following items:
 - I ☒ Basis of the opinion
 - II ☐ Priority
 - III ☒ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
 - IV ☐ Lack of unity of invention
 - V ☒ Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
 - VI ☐ Certain document cited
 - VII ☒ Certain defects in the international application
 - VIII ☒ Certain observations on the international application
3. The applicant is hereby **invited to reply** to this opinion.

When? See the time limit indicated above. The applicant may, before the expiration of that time limit, request this Authority to grant an extension, see Rule 66.2(d).

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also: For an additional opportunity to submit amendments, see Rule 66.4.
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 bis.
For an informal communication with the examiner, see Rule 66.6.

If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.
4. The final date by which the international preliminary examination report must be established according to Rule 69.2 is: **15/10/2001**.

Name and mailing address of the international preliminary examining authority:



European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer / Examiner

Nestby, K

Formalities officer (incl. extension of time limits)

Ipinazar, P
Telephone No. +49 89 2399 8131



I. Basis of the opinion

1. This opinion has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed".*):

Description, pages:

1-5 as originally filed

Claims, No.:

1-10 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

III. Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

1. The questions whether the claimed invention appears to be novel, to involve an inventive step (to be non-obvious), or to be industrially applicable have not been and will not be examined in respect of:

☐ the entire international application,

☒ claims Nos. 4 to 8,

because:

☐ the said international application, or the said claims Nos. relate to the following subject matter which does not require an international preliminary examination (*specify*):

☒ the description, claims or drawings (*indicate particular elements below*) or said claims Nos. 4 to 8 are so unclear that no meaningful opinion could be formed (*specify*):
see separate sheet

☐ the claims, or said claims Nos. are so inadequately supported by the description that no meaningful opinion could be formed.

☐ no international search report has been established for the said claims Nos. .

2. A written opinion cannot be drawn due to the failure of the nucleotide and/or amino acid sequence listing to comply with the standard provided for in Annex C of the Administrative Instructions:

☐ the written form has not been furnished or does not comply with the standard.

☐ the computer readable form has not been furnished or does not comply with the standard.

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N) Claims 1-3 no

Inventive step (IS) Claims 9, 10 no

Industrial applicability (IA) Claims

2. Citations and explanations
see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

Re Items III, V and VIII

Non-establishment of opinion with regard to novelty, inventive step and industrial applicability. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement. Certain observations on the international application

1. Although the subject matter of claim 1 is disguised as pertaining to a bleaching activator, by the wording "the activator **is** a mono-, di- or triformic, acetic or propionic ester of glycerol." (emphasized by the examiner), de facto said chemical compounds are claimed as such.

Said glycerol esters are - of course - known compounds. If the Applicant should have any doubts as to this fact, then attention is drawn to EP-A-0 481 792 (hereafter referred to as D1).

The subject matter of claims 1 to 3 is therefore not novel (Article 33(2) PCT).

2. Since claim 1 is a product claim directed to said glycerol esters as such, the scope of dependant claims 4 to 8 is unclear (Art. 6 PCT) for the following reasons:
 - 2.1 Said features relate to a specific use which does not change the characteristics of the product. Besides, in claim 7 reference is made to a "bleaching solution" which is not mentioned in claim 1.
3. It is known in the art, see WO-A-94/18299 and WO-A-95/21290 (D2, respectively D3) to bleach cellulose pulp with compositions comprising peroxide as well as bleaching activators. It would appear obvious to a skilled person to apply the activators disclosed in D1, e. g. glycerol triacetate, in a bleaching method according to D2/D3, thereby arriving at the subject matters of claims 9, 10 without having to exercise any inventive step.

In view of the paragraph above, the skilled person would regard it a normal design procedure to combine all the features set out in claims 9, 10. Thus, the subject matter of claims 9, 10 does not involve an inventive step and does not satisfy the criterion set forth in Article 33(3) PCT.

Re Item VII

Certain defects in the international application

4. Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1, D2, D3 is not mentioned in the description, nor are these documents identified therein.



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Europäisches
Patentamt

Generaldirektion 2

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Patent Office

Directorate General 2

Office européen
des brevets

Direction Générale 2

Correspondence with the EPO on PCT Chapter II demands

In order to ensure that your PCT Chapter II demand is dealt with as promptly as possible you are requested to use the enclosed self-adhesive labels with any correspondence relating to the demand sent to the Munich Office.

One of these labels should be affixed to a prominent place in the upper part of the letter or form etc. which you are filing.

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner
 US Department of Commerce
 United States Patent and Trademark
 Office, PCT
 2011 South Clark Place Room
 CP2/5C24
 Arlington, VA 22202
 ETATS-UNIS D'AMERIQUE
 in its capacity as elected Office

Date of mailing (day/month/year)
 23 February 2001 (23.02.01)

International application No.
 PCT/FI00/00534

Applicant's or agent's file reference
 50136

International filing date (day/month/year)
 14 June 2000 (14.06.00)

Priority date (day/month/year)
 15 June 1999 (15.06.99)

Applicant

JÄKÄRÄ, Jukka et al

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:

14 December 2000 (14.12.00)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was

☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO
 34, chemin des Colombettes
 1211 Geneva 20, Switzerland

Facsimile No.: (41-22) 740.14.35

Authorized officer

F. Baechler

Telephone No.: (41-22) 338.83.38

REC'D 14 SEP 2001

WIPO


PCT

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

14

| | | |
|---|---|--|
| Applicant's or agent's file reference 50136/JS | FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416) | |
| International application No. PCT/FI00/00534 | International filing date (day/month/year) 14/06/2000 | Priority date (day/month/year) 15/06/1999 |
| International Patent Classification (IPC) or national classification and IPC D21C9/16 | | |
| Applicant KEMIRA CHEMICALS OY et al | | |
| <p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 5 sheets, including this cover sheet.</p> <p><input checked="" type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of 1 sheets.</p> | | |
| <p>3. This report contains indications relating to the following items:</p> <ul style="list-style-type: none">I <input checked="" type="checkbox"/> Basis of the reportII <input type="checkbox"/> PriorityIII <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicabilityIV <input type="checkbox"/> Lack of unity of inventionV <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statementVI <input type="checkbox"/> Certain documents citedVII <input checked="" type="checkbox"/> Certain defects in the international applicationVIII <input checked="" type="checkbox"/> Certain observations on the international application | | |
| Date of submission of the demand 14/12/2000 | Date of completion of this report 11.09.2001 | |
| Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 | Authorized officer Nestby, K Telephone No. +49 89 2399 8625 | |



INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/FI00/00534

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, pages:

1-5 as originally filed

Claims, No.:

1-10 as received on 23/05/2001 with letter of 23/05/2001

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/FI00/00534

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | | | |
|-------------------------------|------|--------|------|
| Novelty (N) | Yes: | Claims | 1-10 |
| | No: | Claims | |
| Inventive step (IS) | Yes: | Claims | 1-10 |
| | No: | Claims | |
| Industrial applicability (IA) | Yes: | Claims | 1-10 |
| | No: | Claims | |

2. Citations and explanations see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. It is known in the art, e. g. from WO-A-94/18299 and WO-A-95/21290 (hereafter referred to as document D2, respectively D3) to bleach cellulose pulp with compositions comprising peroxide as well as bleaching activators. The particular activators of the present invention, however, are not mentioned.

The subject-matter of all claims 1 to 10 is therefore novel (Article 33(2) PCT).

2. Although the use of glycerol triacetate as bleaching activator is described in EP-A-0 481 792 (D1), there is no incentive for the skilled person to use this particular activator in a bleaching composition as disclosed in D1 and/or D2, for the following reasons:

The specific preferred activator used in D3 (closest prior art) is tetraacetylene diamine (TAED). The drawbacks of TAED are discussed in the description of the present application on page 1, line 28 to page 2, line 2.

Said drawbacks are solved by the present invention; besides, a cellulose pulp treated with said composition becomes improved opacity. There is no hint whatsoever in D1 that a detergent composition in tablet form (disclosure of D3) could possibly solve said problems and at the same time bring about an improvement of cellulose pulp opacity properties.

The solution to this problem proposed in claims 1, 6, 7 of the present application is hence considered as involving an inventive step (Article 33(3) PCT). The subject matter of dependent claims 2 to 5, 8 to 10 represents preferred embodiments of the novel and inventive subject matter of claims 1, 7.

Re item VII

Certain defects in the international application

3. Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1, D2, D3 is not mentioned in the description, nor

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/FI00/00534

are these documents identified therein.

4. The description is not in conformity with the amended claims as required by Rule 5.1(a)(iii) PCT.

Re Item VIII

Certain observations on the international application

5. Although claims 1, 7 are drafted in the two-part form the feature "bleaching solution comprises a bleaching activator" is incorrectly placed in the characterising portion, as it is disclosed in documents D1, D2 in combination with the features placed in the preamble (Rule 6.3(b) PCT).

Claims

1. A peroxide bleaching solution for bleaching paper pulp containing lignin, characterized in that said bleaching solution comprises a bleaching activator comprising a mono-, di- or triformic, acetic or propionic ester of glycerol.
2. A peroxide bleaching solution according to Claim 1, wherein the activator is glycerol triacetate.
3. A peroxide bleaching solution according to Claim 1, wherein the activator is glycerol diacetate.
4. A peroxide bleaching solution according to Claim 1, wherein the bleaching solution additionally comprises a chelating agent, a stabilizer, lye, and waterglass.
5. A peroxide bleaching solution according to Claim 1, wherein the bleaching solution comprises peracetic acid.
6. Use of mono-, di- or triformic, acetic or propionic ester of glycerol as a bleaching activator in peroxide bleaching of paper pulp containing lignin to improve the opacity.
7. A method for bleaching pulp containing lignin with a bleaching solution containing hydrogen peroxide, characterized in that a bleaching activator comprising a mono-, di- or triformic, acetic or propionic ester of glycerol is added to the bleaching to improve the opacity.
8. A method according to claim 7, wherein the pulp to be bleached comprises a mechanical pulp.
9. A method according to Claim 7, wherein the activator is added in an amount of 0.2 - 5 kg/ton of pulp.
10. A method according to Claim 7, wherein the activator is added in an amount of 1 - 3 kg/ton of pulp.